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Journal of the European Ceramic Society 25 (2005) 781–791

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Characterisation of the green machinability of AlN powder compacts

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Received 11 November 2003; received in revised form 7 February 2004; accepted 13 February 2004
Available online 20 June 2004

Abstract

The aim of this work is to study the machinability of aluminium nitride green bodies obtained from dry pressing of spray-dried granules. The characterisation of the green machinability of ceramics is not easy. Indeed, to date, no conventional mechanical test able to represent the machining behaviour of green powder ceramic compacts. Therefore, the first target of this work is to determine possible correlations between mechanical properties, microstructure and machinability and to propose suitable tests. Three types of aluminium nitride granules are investigated. Two of theses types of granules are commercial grades using a thermoplastic binder: a conventional grade for pressing and a specific grade especially developed for green machining. The third grade is under development and uses a thermoset binder. From this work it appears that the compacts containing the thermoset binder exhibit simultaneously the highest mechanical properties and the best behaviour at machining. The good machinability has been correlated to a high work of fracture together with a transgranular mode of fracture of the material.

Keywords: Green machining; Machinability; Mechanical properties; Microstructure; Binders; AlN

1. Introduction

Aluminium nitride is an electrical insulator and an excellent thermal conductor. These properties are particularly attractive for electronic applications where thermal management problems are growing. In high-power electronic devices, the aluminium nitride coolers are usually obtained by dry pressing. For such complex parts a near net shape green machining step is highly desirable.

Indeed, the machining of sintered ceramics is time and energy consuming. Moreover, it usually requires specific equipment such as diamond cutting tools. Consequently, this operation is very expensive and can represent up to 80% of the production cost of a ceramic part. Green machining is about 1000 times faster than conventional ceramic machining and can be operated with almost all conventional tools and techniques used for metals.

To reach this target, it is necessary to improve the green body's properties in order to make the machining before sintering efficient. Indeed, the green body properties are often too low and flaws may appear in the part after sintering although nothing was observed just after machining in the green state.

Research in green ceramic machining is rare and know-how is mainly empirical. Many problems remain to be solved to make green machining economically attractive (e.g., clamping devices, or machining chips extraction). One of the most critical point to be investigated is the characterisation of machinability of the green bodies. To date, industrial work is based on experience. It is very difficult to predict if a green body will withstand green machining and the best strategy for performing the work.

In the case of metals, machinability can be defined from the specific resistance against cut of the material, from the tool wear and from the quality of the surface after machining. The rate of material removal is often used to measure machinability: the better the rate of material removal, the better the machinability. For metals and glass ceramics, hardness can also give useful information concerning the machining behaviour. But for the green bodies these criteria cannot be used. The rate of material removal is not the limiting parameter. The study of chip formation shows two major types of behaviour at machining, brittle (case of sintered ceramics) and ductile (case of metals) and it is not easy to classify ceramic green compacts in one of these grades. Several studies about green machining consider the influence of machining parameters, as the cutting speed, the feed

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0955-2219/$ – see front matter
doi:10.1016/j.jeurceramsoc.2004.03.006

rate and the depth of cut, on the cutting force,[1] the surface roughness and the strength of the green body.[2] In the case of compacts pressed from granules,[2] machinability seems better when is set to a cutting mode instead of a spalling mode of granules. The type of the organic additives and the strength of the granules also have a high influence on cutting force. A few authors used tool wear as an indicator of machinability.[3,4] However, the best criterion is the "quality" of the samples after machining. This "quality" is very subjective and difficult to quantify. Scheller[5] notes the number of flakes caused by the milling on the machined green body. Flakes generation is usually considered as a major problem and may lead to scrapping of the part. Edge retention is also used to characterise machinability in the case of green compacts pressed from various zirconia granules.[6,7] The authors show that edge retention is better for samples with low strength and that intergranular fracture is preferable to transgranular fracture. According to Song and Evans,[6] the edge quality is determined by the original critical defect size in the compacts. Therefore, high compaction pressure lowers the edge retention by decreasing the critical defect size. This explanation seems to be adapted to this specific case but the criterion is very subjective and imprecise.

The characterisation of the machinability of the green compacts is of major importance in order to optimise the green body's behaviour during machining.[8] Moreover, there is clearly a need in defining a criterion of machinability, which can be easily and broadly used. For that purpose, different techniques are tested in this work to measure the machinability. A correlation between machinability tests, the mechanical properties and the microstructure of the green compacts is proposed.

2. Experimental methods

2.1. Preparation of the green bodies

2.1.1. Granules composition

The raw material used in the present work is an aluminium nitride powder commercialised by Atofina (France) and named Pyrofine A. This AlN powder is spray-dried with organic additives (plasticiser, binder) to prepare granules suitable for dry pressing. Three compositions are investigated in this work.

Two of these are commercial grades (further called granule's types A and B) containing a thermoplastic binder (Polyethyloxazoline PEO, $M = 50000$ g/mol, $T_g = 70\,^{\circ}$C) supplied by The Dow Chemical Company. The difference between these two compositions is the amount of binder (Table 1).

The last grade (granule's type C) is based on a thermoset binder. With this new composition, the target is to improve the mechanical properties of the green bodies thanks to the in situ cross-linking of the binder during the shaping. The system investigated is a polyester resin (Palatal A 400-01, $T_g = 121\,^{\circ}$C, supplied by DSM BASF) as binder with a peroxide type catalyser (Luperox P from Elf Atochem Deutschland).

In both case PEG 400 is used as plasticiser. The detailed compositions of the spray-dried powders are given in Table 1.

2.1.2. Specimens forming

Green body's specimens were shaped with a single axe double action press. In order to determine a compaction pressure suitable to prepared specimens for machining tests, the compaction behaviour of the granules was observed by SEM for different pressures. Next to this study, test bars for mechanical characterisation (52 mm × 8.5 mm × 5.5 mm) and for machining tests (54 mm × 50 mm × 8 mm) were prepared by compaction of the granules under 100 MPa. The compacts obtained from granule's type C were heated 1 h at 160 °C after shape forming.

2.2. Characterisation of the mechanical properties

2.2.1. Young's modulus, strength and work of fracture

The three types of green body's specimens (further called compacts A, B and C) were tested by 4-point bending on a Schenck Trebel machine. The testing parameters were: a cross-head speed of 0.1 mm/min and the spans l = 10 mm and L = 35 mm. During this test the mechanical stress is applied to the sample with an imposed strain as it is the case during a machining test.

The green strength (σ_r) and an estimation of the Young modulus (E) are calculated from the load–displacement curves. Relations used for the calculations are:

$$\sigma_r = \frac{3F_{max}(L-l)}{2w^2b},\ E = \frac{\Delta F}{\Delta y}\frac{(L-l)^2}{4bw^3}(L+2l)$$

Table 1
Granules formulations

Nature	Component	Granules (wt.%/AlN)		
		A (B9F2)	B (B9F7)	C (MD8)
Ceramic	AlN	100	100	100
Sintering aid	Y_2O_3	5	3	5
Dispersant	Phosphoric ester	0.5	0.5	0.5
Binder		4 PEO	7 PEO	3.5 Polyester resin
Plasticiser	PEG 400	0.7	0.7	0.9
Lubricant	Stearic acid	0	0.2	0

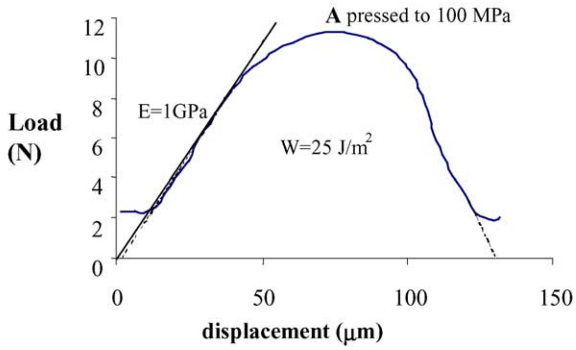

Fig. 1. Flexure curve used to calculate σ, E and W.

where b and w are the sample width and height respectively. y is the deflection under loading rolls, F_{max} is the maximum load.

The work required to break the compact (work of fracture W_{1C}) is also calculated from the area under the curve load–displacement (Fig. 1).

2.2.2. Toughness

Toughness (K_{IC}) was obtained by 4-point flexure tests on notched beams (SENB tests). The notch depth was about 2.5 mm ($a/w \approx 0.3$). Due to the very low level of loading, the samples were loaded by drop by drop of water. Toughness was obtained from the relation:

$$K_{IC} = Y\sigma\sqrt{a}$$

where a is the notch depth and w is the sample height. Y is a geometric factor given by:

$$Y = 1.99 - 2.47\frac{a}{w} + 12.97\left(\frac{a}{w}\right)^2 - 23.17\left(\frac{a}{w}\right)^3 + 24.8\left(\frac{a}{w}\right)^4$$

The preparation of green body's specimens for SENB tests is not easy due to the difficulties to realise suitable notches. Therefore, the toughness of the samples was also measured from scratch tests. In these tests, a Vickers diamond was introduced in the material with a normal stress, F_n, lower than 10 N. Then the compact was moved linearly to form a groove until the diamond reached the edge. Near the edge, a flake was formed (Fig. 2). The size of this flake, l_c, can be correlated to the toughness according to the relation:[9]

$$K_{IC} = 0.0123 F_n l_c^{-3/2}$$

2.2.3. Edge flaking

The "edge flaking" is a test usually performed to measure the resistance to flake of brittle materials.[10] This characteristic was also measured and correlated to the machinability of green compacts. A Vickers diamond was introduced in the sample at a distance d of the edge and a normal stress continuously increasing until a flake was formed. The stress F required to form the flake was plotted versus the distance d. The edge toughness M was given by the slope of the straight line F versus d (Fig. 3).

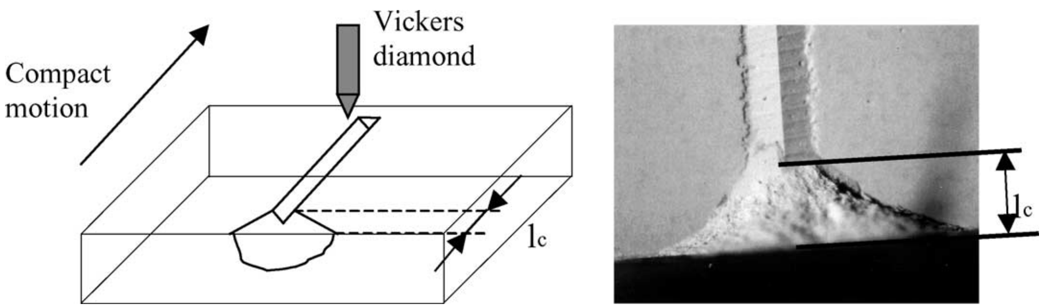

Fig. 2. Scratch test.

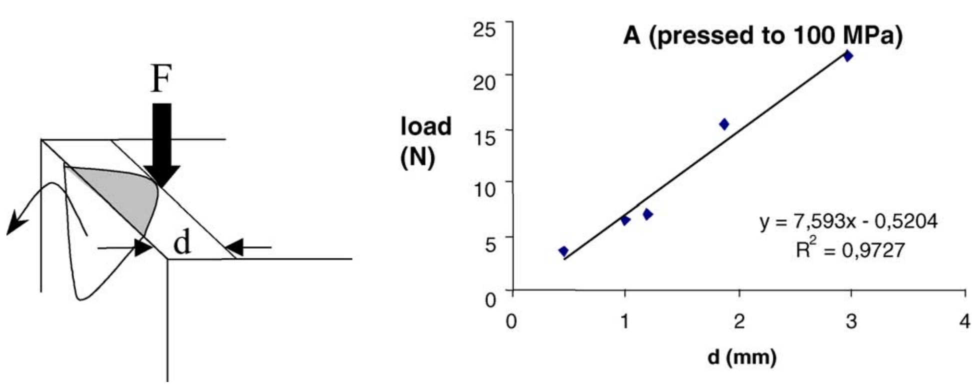

Fig. 3. Edge flaking.

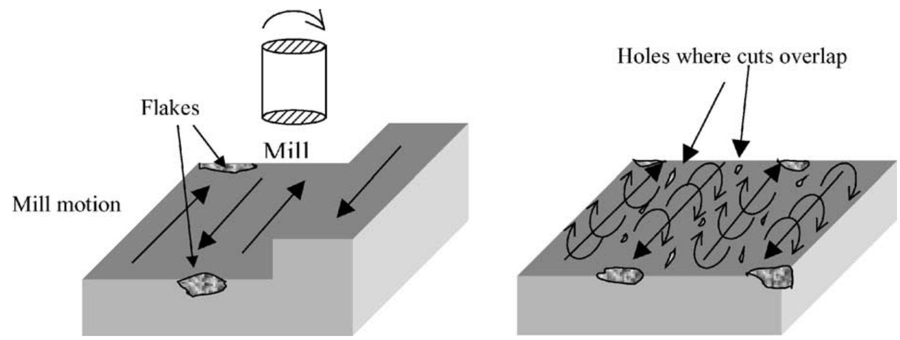

Fig. 4. Milling tests.

2.3. Machining tests

2.3.1. Milling tests

A milling test was used as an indication of machinability. The machining was performed on a Dufour universal milling machine with a 20 mm diameter mill. The mill was fitted with two tips of TiCN cermet (Tungaloy, Toshiba). The tip was replaced every five samples. The samples were maintained on the machine with a vacuum system. The milling conditions were: a cutting speed of 900 rpm giving a 56.6 m/min tip speed and a feed rate of 375 mm/min giving a feed of 0.21 mm per tooth.

The surface of the sample was machined by four cuts with a depth of cut of 3.5 mm (Fig. 4). These severe machining conditions were chosen to drastically stress the samples. The aim of these tests was to create flakes when the mill goes out of the sample.

The volume of flakes was taken as an indication of the machinability of the sample. This volume was calculated from the difference between the theoretical volume of the part after machining and the volume really measured (from the ratio weight/density).

2.3.2. Scratch test

Another way to evaluate machinability of green compacts is the "scratch test". A groove, 1 mm deep, was created at the surface of the material by displacement of a carbide tip at a constant speed of 375 mm/min. These tests were performed on the milling machine. Characterisation of the machining behaviour was made from the number of the flaws in the groove and from the surface and features of the flakes around the groove (Fig. 5). These parameters were quantified from photographs.

2.4. Microstructural characterisation

Observations of fracture surfaces by SEM can give interesting information about the mode of fracture: transgranular or intergranular. Binder and porosity distributions were also characterised by optical microscopy and SEM. The green body's homogeneity is a parameter that must be taken into consideration when investigating the machining behaviour. The chips collected during machining were observed by optical microscopy and SEM. The machined surface and the flakes surface were also examined by microscopy.

3. Experimental results

3.1. Compaction of the granules

SEM observations of the surface of compacts A are given (Fig. 6) for different compaction pressures. Voids at the interfaces between granules can easily be observed, even after forming at high pressure (380 MPa).

A comparison of the surfaces of the compacts obtained from the three types of granules under a compaction pressure of 100 MPa shows that compacts B are similar to compacts A and clearly exhibit voids between granules (Fig. 7). On the contrary, these interfaces are not observed in the case of granules C.

From this result a pressure of 100 MPa was chosen to prepare test bars for mechanical and machining test. Indeed, 100 MPa leads to a good compaction of granules C (disappearance of the interfaces) and an higher pressure do not really improve the compaction of granules A and B.

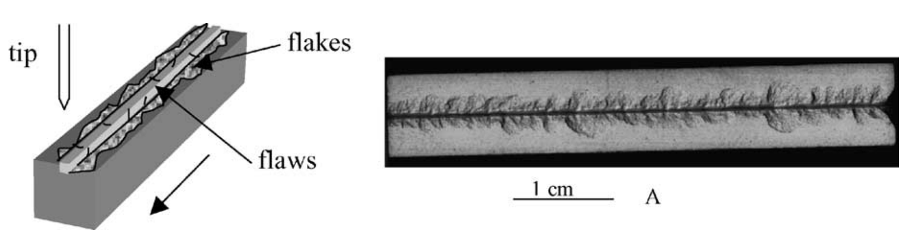

Fig. 5. Scratch tests.

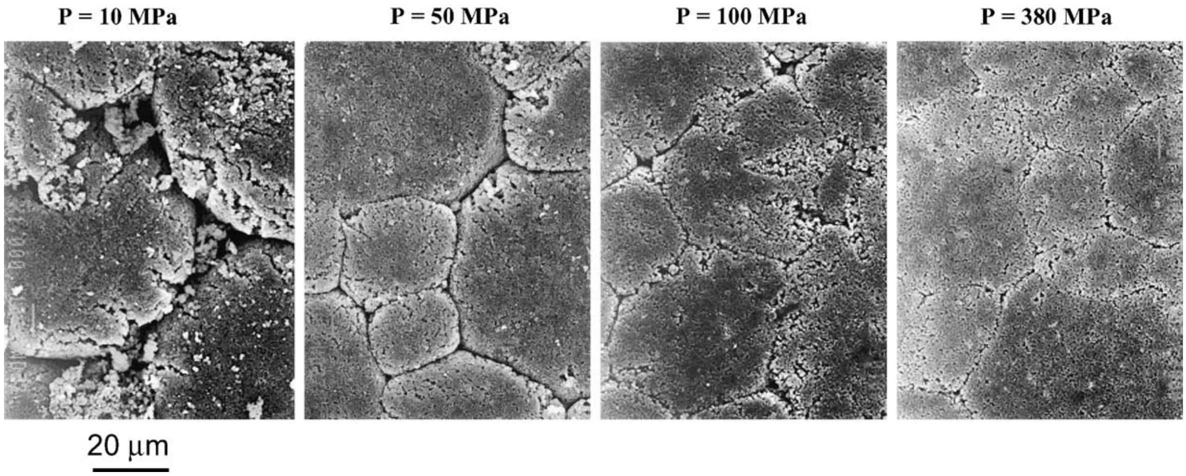

Fig. 6. Surfaces under upper punch at different forming pressures for granules A.

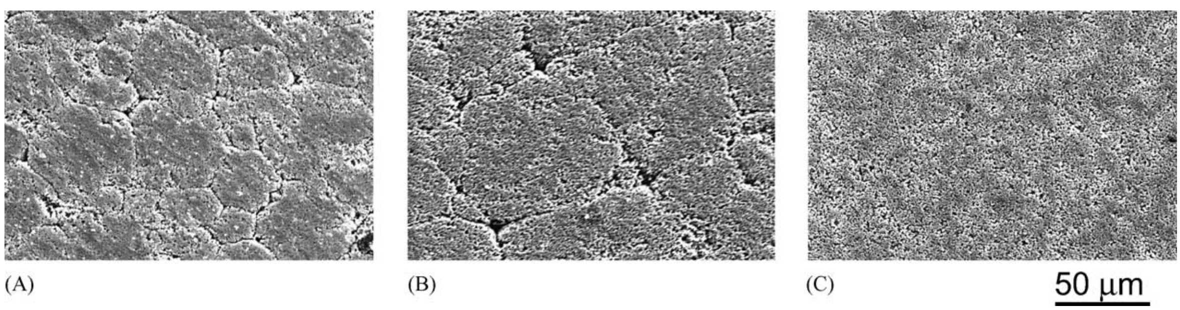

Fig. 7. Surfaces under upper punch of the three types of granules pressed to 100 MPa.

Table 2
Mechanical characterisation of the green compacts

	σ_r (MPa)	E (GPa)	K_{IC} (kPa m$^{1/2}$)	Work of fracture (J/m^2)	M-edge flaking (N/m)	HV (MPa)
A	1.28	0.8	44.9	18	7.7	2.8
B	2.74	0.5	53.9	100	9.4	4.4
C	3.41	0.7	92	165	25.2	6.4

3.2. Mechanical properties

The mechanical properties measured on the three types of compacts are significantly different (Table 2).

With the same a compaction pressure of 100 MPa, the strength of the green compacts C is nearly three times that of the green compacts A. The stress intensity factor K_{1C} and the work of fracture W_{1C} are also much higher (respectively 2 and 10 times higher). The strength and the work of fracture of compacts B are near that of compacts C but the toughness remains as low as that of compacts A.

The mechanical behaviour of the three compacts is also different. Indeed, as show the load–displacement curves (Fig. 8), the rupture of green compacts C occurs suddenly once the maximum load reached ($\varepsilon_r \approx \varepsilon_i$). On the contrary, with compacts A or B the fracture occurs for a deformation ε_r significantly higher than the deformation ε_i corresponding to the maximal stress. For these latter compacts, the cracks propagate slowly after the maximal stress (Fig. 9).

All the curves show a significant non-linear loading that can be attributed to a plastic deformation.

Weibull statistical analysis (Table 3) has been performed with 20 samples of the three types of green bodies. The Weibull plots are shown in Fig. 10. No significant

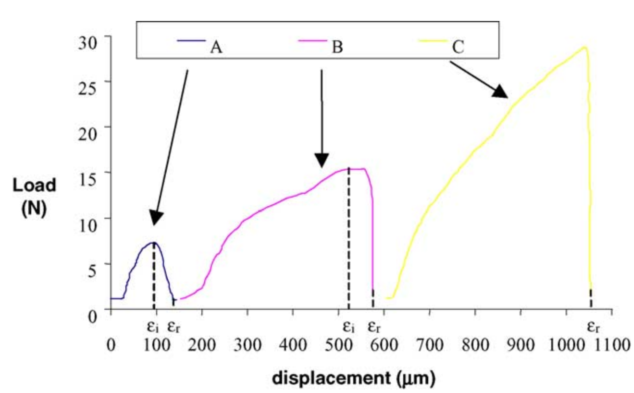

Fig. 8. Curves obtained by 4-point bending tests.

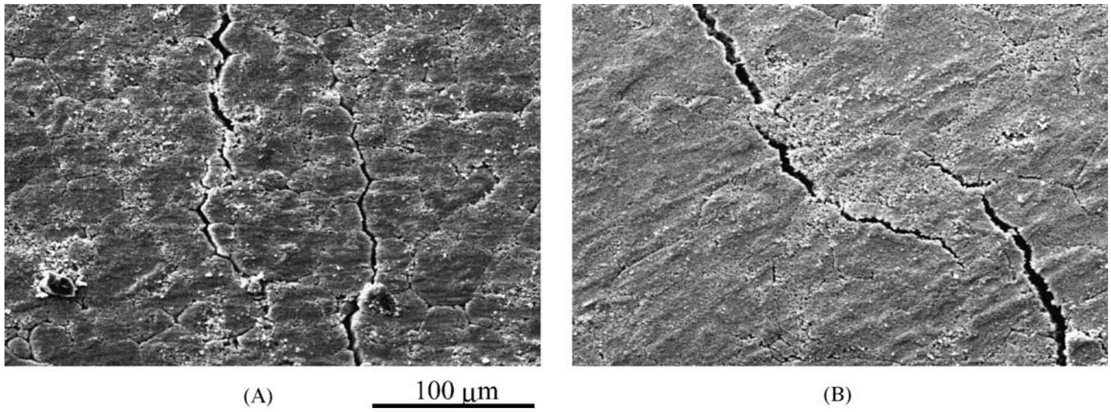

Fig. 9. Propagation of the flaw after F_{max}.

Table 3
Weibull parameters and average strengths

	m	σ_0 (MPa)	Average strength (MPa)	Standard deviation
A	11.9	1.35	1.28	0.13
B	12.8	2.85	2.74	0.23
C	9.4	3.50	3.41	0.38

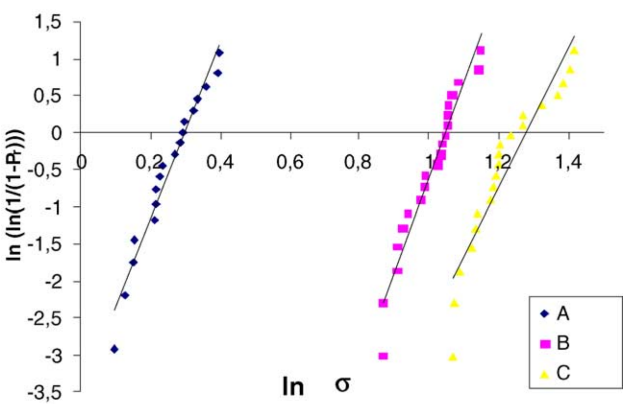

Fig. 10. Weibull statistic.

differences are found between the m values for the different green compacts.

Remark

The thermoplastic binder used in granules A and B is very hygroscopic. Consequently, the glass transition temperature of the binder changes drastically with moisture. The mechanical characterisation of these compacts requires a severe control of test and storage conditions. This is especially the case for granules B, which contain a high amount of the thermoplastic binder (7%).

3.3. Microstructures

SEM observations of the fracture surface after mechanical testing shows significant differences in the mode of fracture according to the composition of the specimens (Figs. 11–13). The surface of the green compact A shows a majority of intergranular fracture. On the contrary, the fracture of green compacts B is mainly transgranular. For green compacts C, the way of fracture is completely transgranular.

These results can be correlated with the SEM observations of the granules after the compaction (Fig. 7). An intergranular mode of fracture (compacts A and B) is linked

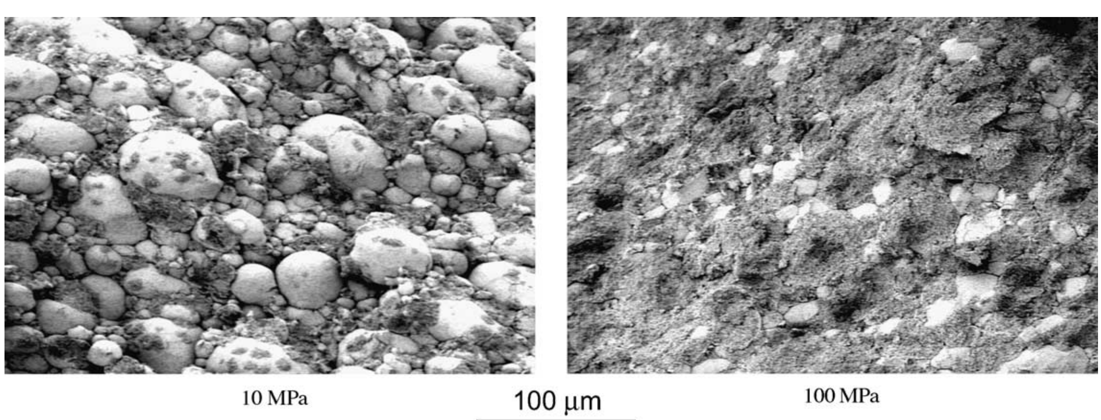

Fig. 11. Fracture of green bodies A pressed to 10 and 100 MPa.

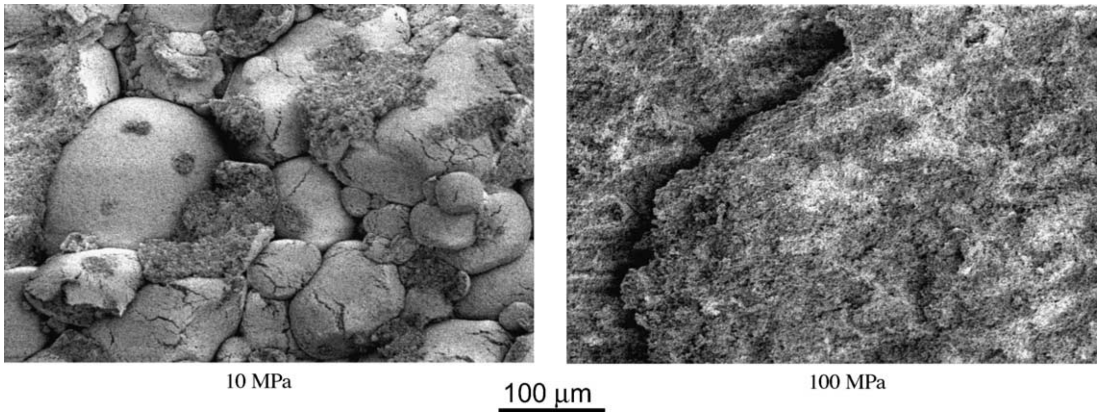

Fig. 12. Fracture of green bodies B pressed to 10 and 100 MPa.

to a low cohesion between the granules corresponding to the presence of remaining voids at the interfaces.

The distribution of the binder inside the green body has also been characterised from a specific method consisting in a heat treatment of the samples before observations by optical microscopy.[11] The heat treatment is chosen to start the pyrolysis of the binder and so to reveal it by its colour. Green compacts A and B were heated to 300 °C. A high binder concentration around granules is seen on the micrographs (Fig. 14), especially for granules B which contain a high amount of binder (7%). This binder rich layer is due to the migration of the organic additives during spray drying. It strongly increases the cohesion between granules and for granules B, this cohesion becomes higher than the cohesion inside the granules. This explains the more transgranular mode of fracture in comparison to granules A. Granules C were heat treated at 350 °C. The organic additives distribution is much more homogeneous than for granules A and B and no granules interfaces are observed.

3.4. Machinability

The volumes of flakes (measured as defined in Section 2.3) formed during milling tests for the three types of green bodies are shown in Fig. 15. This volume is only 100 mm^3 for compacts C in comparison with about 200 and 400 mm^3 for compacts B and A, respectively.

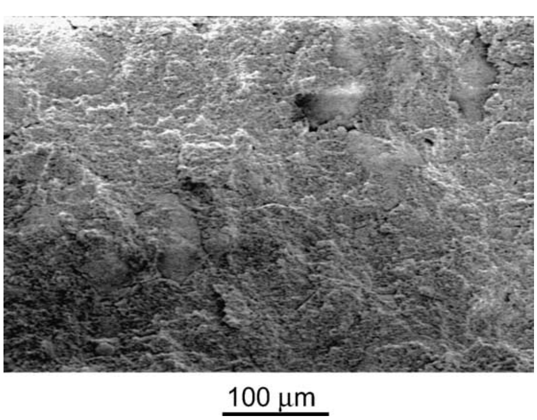

Fig. 13. Fracture of green bodies C pressed to 10 MPa.

The observations of the surface of the samples after machining confirm these results (Fig. 16). The surface of compacts C shows no hole at the cuts overlap and the flakes volume is clearly smaller in comparison to compacts A and B.

On the contrary to the volume of flakes, the differences between the surface shown by compacts A and B are not obvious.

The surfaces of the flakes formed during machining were observed (Fig. 17). As for the fracture surfaces during mechanical tests, the fracture is intergranular for green compacts A and fully transgranular for green compacts C.

The scratch tests performed on the three types of green bodies reveal different behaviours (Fig. 18).

- Observations inside the grooves show a high density of flaws for green compacts B. This density of flaws is lower for green compacts A. On the contrary, no flaw is observed in the case of the green compacts C.
- The characteristics of the flakes along the grooves show also differences between the compacts. For green bodies A, the flakes are large (>2 mm from the mean line of the groove). The rough surface of the flakes is due to an intergranular mode of fracture. Green compacts B also show a high relief of the fracture surface but the size of the flakes is less (1–2 mm from the mean line of the groove). In the case of compacts C, the size is about the same than with compacts B but the smooth surface is linked to a transgranular mode of fracture.

4. Discussion

First, a classification of the machinability of the green compacts can be proposed from the measurement of the volume of flakes generated during the machining tests.

- A flakes volume lower than 100 mm^3 is linked with few holes on the machined surface. In that case the machinability is considered as good.

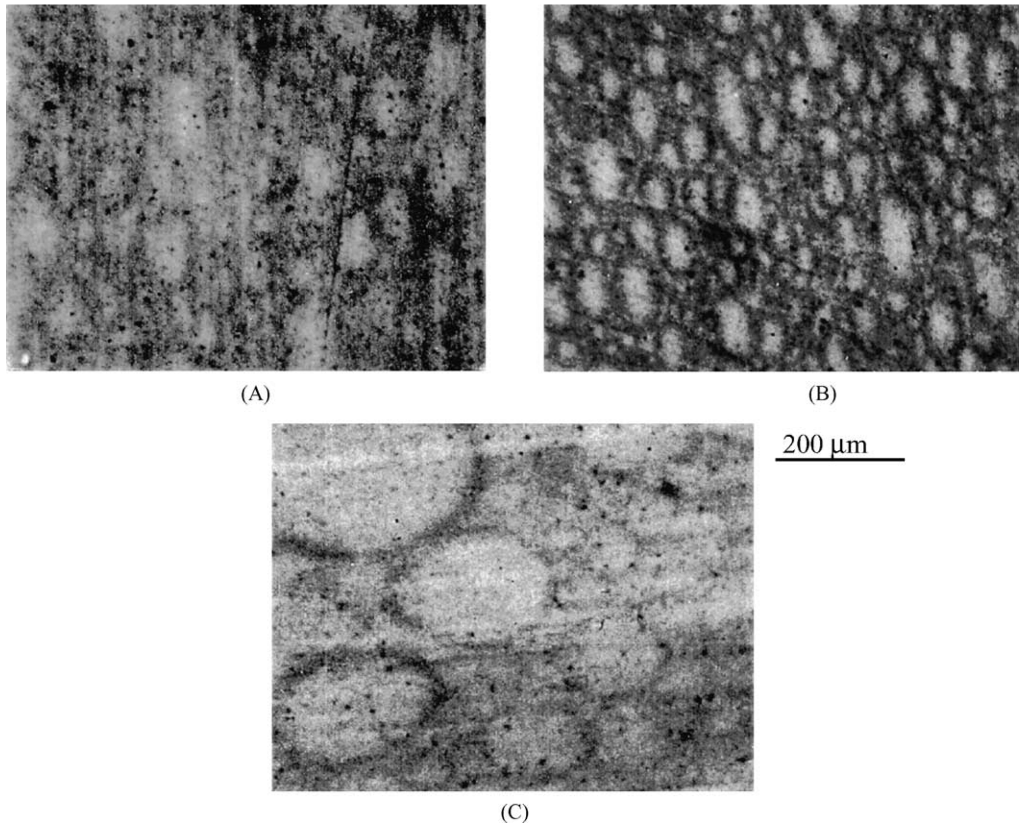

Fig. 14. Observation by optical microscope (dark field) of polished surfaces after heat treatment.

– A flakes volume in the range 100–250 mm^3 is an average result. The behaviour at machining is considered as acceptable.
– A flakes volume higher than 250 mm^3 corresponds to a very bad quality of the machined surface (numerous holes). Machinability is poor.

The machinability is linked to the microstructure and mechanical properties. Two major behaviours can be distinguished.

– First, a good behaviour at machining that can be associated to a brittle machining mode. This is the case with compacts C obtained from granules containing a thermoset binder. The fracture during bending tests is sudden even if plastic strain occurs before the sample breaks. The strength and the work of fracture are high (respectively, >4 MPa and >160 J/m^2). The relief of the fracture surfaces is low, the fracture being fully transgranular. This later point is the result of a high homogeneity in the sample (disappearance of the granules).
– Second, a bad machinability corresponding to the spalling of the granules during machining. This is typically the behaviour obtained with green compacts A prepared from granules containing a thermoplastic binder. The fracture is controlled, flaws propagate slowly after the maximum stress has been reached, but the strength and the work of fracture are very low. The relief of the fracture surfaces is significantly higher than for granules C due to a mode of fracture being mainly intergranular. In this case the granules do not totally disappear after compaction and a layer of binder is founded around the granules forming an easy route for crack propagation.

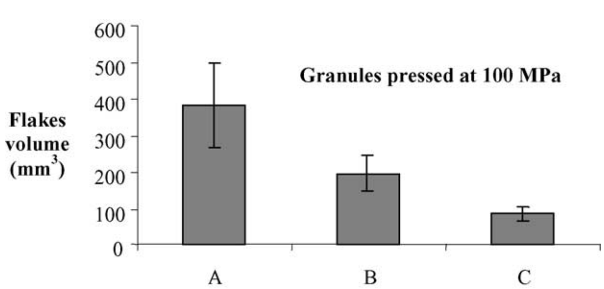

Fig. 15. Results of the milling tests for the three types of green compacts.

In the case of green compacts B, the increase in amount of thermoplastic binder strengths the cohesion between the granules and lead to a mainly transgranular mode of fracture. The work of fracture is significantly higher than with compacts A and the machinability is better. In fact, the mechanical behaviour of green compacts B is near that of green compacts C (high work of fracture, almost sudden fracture).

Although the tests where performed with different rates, the results of the scratch tests are in good agreement with

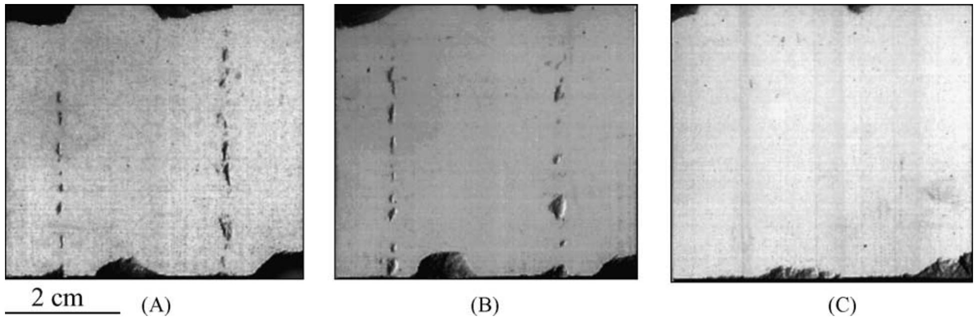

Fig. 16. Samples after machining.

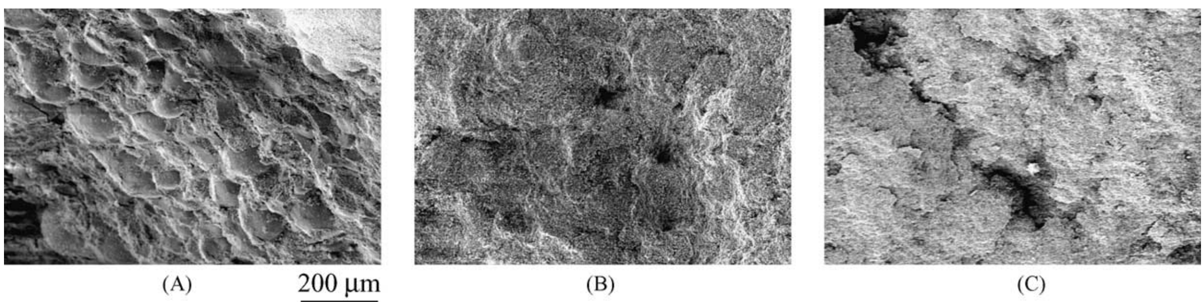

Fig. 17. Mode of fracture during machining—surfaces of the flakes.

the machining tests and can be an easy way to characterise the machinability:

- For green bodies C, flakes are observed around the groove and no flaw is found inside the groove. The roughness of the surface of the flakes is low. These results correspond to a brittle machining mode ensuring a good machinability.
- On the contrary, for green compacts A and B, the numerous flaws are observed along the groove and the roughness of the flakes is higher. This is typical of plastic damage associated with an intergranular mode of fracture.

To summarise (Table 4), if a good machinability is defined as a low volume of flakes, then the good machinability can be associated to:

- A good homogeneity of the green bodies (disappearance of the granules interfaces) leading to a transgranular mode of fracture.

Table 4
Characterisation of the green machinability

Machinability	Good	Medium	Bad
Volume of flakes (mm^3)	<100	$100 < V < 250$	>250
Other criteria	$\sigma > 4\,MPa$ $W_{1C} > 160\,J/m^2$ $K_{1C} > 100\,kPa\,m^{1/2}$	$\sigma > 1.5\,MPa$ $W_{1C} > 100\,J/m^2$ $K_{1C} > 50\,kPa\,m^{1/2}$	$M < 10\,N/m$
Fracture	Transgranular	Transgranular and intergranular	Highly intergranular
Porosity	High but homogeneous	Intergranular porosity	Intergranular porosity
Microstructure	Total disappearance of the granules Uniform layout of the binders	Adhesion between granules > internal cohesion More binder on the surfaces of the granules	Internal cohesion > adhesion between granules More binder on the surfaces of the granules
Machining mode	Brittle	Party ductile	Spalling of granules
Scratch tests	Few flaws	Many flaws	Flaws
Bending curves	Sudden fracture	Controlled fracture	Controlled fracture
Example	C	B	A

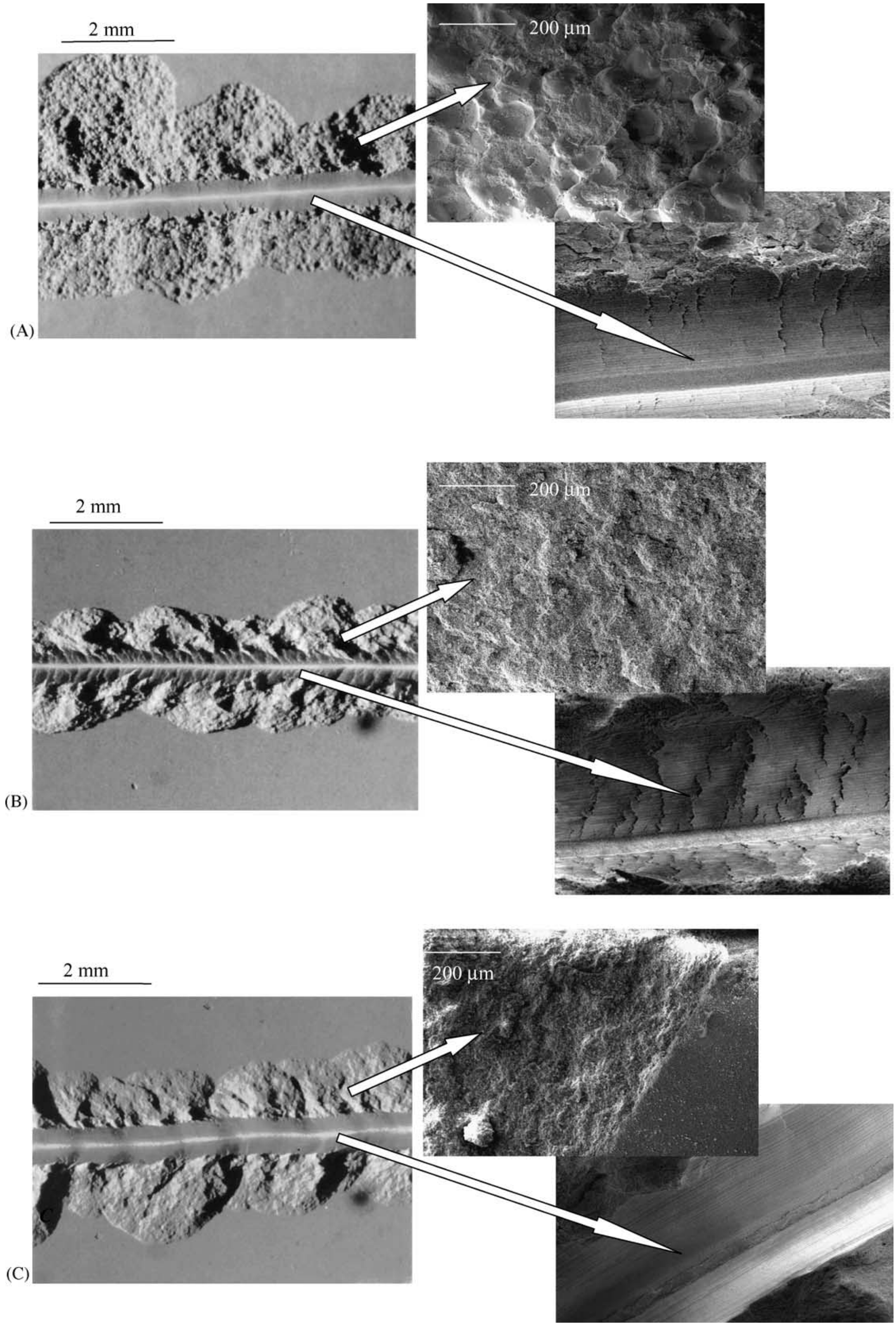

Fig. 18. Results of scratch test.

– A high strength (> MPa) and work of fracture (>160 J/m^2) associated to a sudden rupture during bending tests.

5. Conclusions

This work underlines the differences in behaviour at machining of green AlN compacts with respect to the formulation of the spray-dried granules. In this case, the formulation using a thermoset binder gives a better machinability than with the formulations using a thermoplastic binder. These differences are explained from the microstructure and the mechanical properties of the green compact.

The thermoset binder leads to high homogeneity of the green compacts. Granules totally disappear after compaction, consequently the mode of rupture is fully transgranular. Moreover, the strength measured by bending test is high and the rupture is sudden. These properties lead to brittle machining mode forming a low amount of small flakes. No hole is observed at the cuts overlapping.

On the contrary, the thermoplastic binder forms a layer around the granules, which does not totally disappear after compaction. In this case the mode of rupture is partly or totally intergranular depending on the amount of binder. The strength measured by bending test is much lower and the rupture is controlled. Cracks propagate slowly after the maximal stress has been reached. Consequently, machining leads to the spalling of the granules. The amount of flakes is high and holes are observed at the cuts overlap.

Finally, the study of the mechanical properties and of the microstructure of green compacts is a mean to predict the green machinability. Some threshold values can be given in order to obtain a low volume of flakes and then good machinability. A good behaviour is obtained if the fracture of the compact during a bending test is sudden and the work of fracture is higher than 100 J/m^2.

Acknowledgements

The authors are grateful to the Région Rhône-Alpes for supporting this research by a grant and to ATOFINA for supplying AlN powder and taking part in the study.

References

1. Klocke, E., Gerent, O. and Schippers, C., Machining of advanced ceramics in the green state. *Ceram. Forum Int.* 1997, **74**(6), 288–290.
2. Maier, H. R. and Michaeli, N., Green machining of alumina. *Key Eng. Mater.* 1997, **132–136**, 436–439.
3. Konig, W. and Wagemann, A., Machining of ceramic components: process-technological potentials. Proceedings of the International Conference on Machining of Advanced Materials, 20–22 July, 1993, U.S., Gaithersberg, *NIST Special Publication* 1993, (847), 3–16.
4. Janasovits, U., Grothe, A., Pohlmann, H. J. and Lang, G., Improvement of green machining process of Si_3N_4. *Ber. Deutsche Keramishe Gesellschaft* 1999, **76**(5), 24–28.
5. Scheller, W. L., Conventional machining of green aluminum/aluminum nitride ceramics. *Ohio J. Sci.* 1994, **94**(5), 151–154.
6. Song, J. H. and Evans, J. R. G., On the machinability of ceramic compacts. *J. Eur. Ceram. Soc.* 1997, **17**, 1665–1673.
7. Birkby, I., Dransfield, G. P., McColgan, P., Song, J. H. and Evans, J. R. G., Factors affecting the machinability of fine ceramic powder compacts. *Br. Ceram. Trans.* 1994, **93**(5), 183–186.
8. Sheppard, L. M., Green machining—tools and considerations for machining unfired ceramic parts. *Ceram. Ind.* 1999, 65–76.
9. Westerheide, R., Kellen, C., Drüsedau, K. A., Hollstein, T., Dietz, M. and Bühling, L., Charakterisierung und Bewertung von Grünkörpern als Qualitätssichernde Massnahme bei der Grünbearbeitung. In *Werkstoffwoche'96*, May 1996, Symposium 6, Stuttgart, pp. 649–654.
10. McCormick, N. J. and Almond, E. A., Edge flaking of brittle materials. *J. Hard Mater.* 1990, **1**(1), 25–51.
11. Baklouti, S., Chartier, T. and Baumard, J. F., Binder distribution in spray dried alumina agglomerates. *J. Eur. Ceram. Soc.* 1998, **18**, 2117–2121.